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Shear bond strength of brackets to demineralize enamel after different pretreatment methods

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Original Article

Shear bond strength of brackets to demineralize enamel after different pretreatment methods

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ABSTRACT

Objective: To compare the influence of demineralized and variously pretreated demineralized enamel on the shear bond strength of orthodontic brackets.

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Conclusion: Pretreatment with the infiltrating resin is a beneficial approach to increasing the shear bond strength of brackets to demineralized enamel. (*Angle Orthod.* 0000;00:1–6.)

KEY WORDS: Shear bond strength; Brackets; Demineralized enamel

INTRODUCTION

The highest debonding rates of orthodontic brackets occur shortly after placement of the brackets or at later stages of orthodontic treatment, when the brackets have been stressed by mechanical and thermal impacts.[1] Especially in later stages of orthodontic treatment, enamel demineralization adjacent to brackets is an undesired but frequent event and is a matter of concern for the orthodontist.[2]

If this happens, further demineralization of enamel by preventive measures or debracketing of the teeth should be considered, but particularly during later stages of complex treatment, orthodontists often tend to complete the treatment and to rebond the bracket. When rebonding is taken into consideration, it seems advisable to stabilize and protect the first enamel lesions before reapplication of brackets.

Early studies of Robinson et al. and Davila et al.[3,4] described infiltration of carious lesions with organic resins, demonstrating a reduction in pore volume following application of resorcinol-formaldehyde resin. A reduction in pore volume after impregnation of caries-like lesions had already been described.[4–6]

A new approach in treating incipient caries lesions by an infiltration technique was introduced recently.[7]

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Infiltration of caries lesions with low-viscosity light-curing resins is considered a treatment option for noncavitated lesions not expected to arrest or remineralize. In contrast to the conventional sealing concept, wherein a resin layer is glued onto the lesion surface, caries infiltrants penetrate the porous lesion body of initial caries lesions.[8] Caries infiltrants are optimized for rapid capillary penetration and exhibit very low viscosity and high surface tension.[9] Thus, laboratory experiments showed significantly deeper penetration of infiltrants into the lesion body than is seen with conventional adhesives.[8–10] Clinical follow-up studies have proved this concept to be more effective in stopping progression of a carious lesion within 1.5 years of observation, as compared with fluoridation measures.[11] The infiltration technique offers an option to mask labial enamel white spot lesions, rendering those lesions less visible.[12]

It is conceivable that the so-treated carious enamel is provided with increased stability. Remineralization and stabilization of incipient lesions under the support of fluorides is another frequently used method. In this way, white spot carious lesions might be remineralized as the result of incorporation and precipitation of salivary minerals, thus at least arresting the lesion.[13] In case of pretreatment of initial enamel lesions with an infiltrant or fluorides, the question arises—How far do these pretreatments affect bracket bond strength in the pretreated areas?

Numerous studies have used bracket shear bond strength attached to sound enamel for testing of different adhesives.[14–17] Few studies are available showing reduced bracket shear bond strength on fluorosed enamel.[18–20] However, no literature has been found on bracket bond strength with demineralized dental hard tissue. Moreover, at the moment, it is not known how far pretreatment of initial enamel lesions with fluorides or the infiltration technique is beneficial in increasing bracket bond strength to demineralized enamel.

Therefore, this in vitro study evaluated the influence of demineralized and variously pretreated demineralized enamel on shear bond strength of orthodontic brackets. The null hypothesis was that pretreatment with fluorides or an infiltrating resin does not improve bracket bond strength to demineralized enamel.

MATERIALS AND METHODS

Specimen Preparation

The crowns of 60 bovine permanent mandibular incisors from 3-year-old cows were separated from roots, cleaned of periodontal tissue with scalers, and stored in 0.5% chloramine-T solution for a maximum of 7 days. Subsequently, they were placed in distilled water for 6 months maximum at 5°C. The teeth were embedded with the labial surface downward in self-curing acrylic resin (ScandiQuick, Scandia, Hagen, Germany) in cylindrical molds (25 mm diameter; UnoForm, Struers, Bellerup, Denmark).

Embedded teeth were ground flat in a polishing device (LaboPol-21, Struers) under water cooling with paper of P400 grit to expose the enamel and create a smooth parallel surface. The specimens were ultrasonically cleaned (Aquaclean 3, Degussa, Hanau, Germany) and were stored in distilled water between and after the polishing steps.

The 60 samples were randomly assigned to five groups ($n = 12$). Pretreatment and application of products were conducted according to manufacturers' instructions (Table 1).

Specimens in groups 2, 3, 4, and 5 were demineralized by applying the protocol of Buskes et al.[21] (21 days, 37°C). After pretreatment, all specimens were stored in artificial saliva (56 hours, 37°C), mixed according to Klimek et al.[22] The artificial saliva was renewed every 8 hours.

Application of Brackets

On all specimens, stainless steel brackets for central lower incisors (Discovery, slot 0.56 × 0.76 mm/22 × 30, Dentaurum, Ispringen, Germany) with an average surface area of the bracket base of 8.71 mm^2 were attached.

The teeth were etched (35% phosphoric acid gel, 30 seconds), washed with water, and dried by air-blow. The primer of the Transbond XT (3M Unitek, Landsberg, Germany) bracket luting system was applied to the etched surface. Then, the luting material was applied to the bracket base, and the bracket was placed on the tooth with a standardized load of 500 g. Careful removal of excess material was performed with foam pellets and spatula using a loop (4.8× magnification).

In all specimens, light curing was performed for 60 seconds (15 seconds from cervical, incisal, mesial, and distal directions; Epilar Freelight II LED, 1000 mW/cm^2, 3M ESPE, Seefeld, Germany). Finally, the specimens were stored in distilled water (37°C, 24 hours).

Shear Bond Strength Testing

Shear bond strength was tested with a universal testing machine (Z010, Zwick, Ulm, Germany). Plastic cylindrical carriers with embedded teeth and brackets were mounted on a joint and aligned in the testing apparatus, ensuring consistency for the point of force application and the direction of the debonding force. The direction of the debonding force was parallel to the enamel surface in an occlusogingival direction. A

Table 1. Materials and Application Protocols Used in the Present Investigation

Group	Step/Material	Manufacturer	Composition	Lot No.	Application
1	Etchant Gel	Vita Zahnfabrik (Bad Säckingen, Germany)	35% phosphoric acid	99931	Application (30 s), rinsing with water (40 s), air-dry
	Transbond XT	3M Unitek (Landsberg, Germany)	Transb. XT Primer: Bis-GMA, TEGDMA, 4-dimethylamino-benzene ethanol, campherqui-none, hydroquinone Transb. XT luting material: Bis-GMA, bis-EMA, acrylate, monomers, filler	9FP/9GG	Application of thin coat on enamel (primer) and luting mate-rial on bracket base, positioning and excess removal
2	Etchant Gel	As group 1	As group 1	99931	As group 1
	Transbond XT	As group 1	As group 1	As group 1	As group 1
3	Elmex Fluid	GABA (Basel, Switzerland)	1.23% fluoride (as Olaflur and Dectaflur) saccharine, vanillium, aromatica	83371B	Evenly over treatment area
	Etchant Gel	As group 1	As group 1	As group 1	As group 1
	Transbond XT	As group 1	As group 1	As group 1	As group 1
4	Clinpro White Varnish	3M Unitek (Landsberg, Germany)	2.23% fluoride (NaF), alcohol-based solution of modified resins	M12702	Evenly over treatment area
	Etchant Gel	As group 1	As group 1	As group 1	As group 1
	Transbond XT	As group 1	As group 1	As group 1	As group 1
5	Icon	DMG (Hamburg, Germany)	Icon-Etch: hydrochloric acid, pyrogenic silicic acid, surface-active substances	621424	Application (120 s), rinsing with water (30 s), air-dry
			Icon-dry: 99% ethanol		Application (30 s), air-dry
			Icon-Infiltrant: methacrylate-based resin matrix, initiators, additives		Application (180 s), light-curing (60 s), reapplication (60 s), light-curing (40 s)
	Etchant Gel	As group 1	As group 1	As group 1	As group 1
	Transbond XT	As group 1	As group 1	As group 1	As group 1

stainless steel rod with a chisel configuration was used for bracket debonding. Crosshead speed was 1 mm/min. Load at failure was recorded and shear strength values were calculated according to the following equation: $S = F/A$, where S is shear bond strength, F is load at failure (N), and A represents the adhesive area (mm^2).

For fracture analysis, the debonded areas were examined with a stereomicroscope (40× magnification; M3B, Wild, Heerbrugg, Switzerland). Failure was considered as follows: adhesive if the cement/resin was dislodged from enamel; cohesive in cement/resin if the fracture occurred only in cement/resin; and cohesive in enamel if the fracture occurred only in enamel.

Data Analysis

The Statistical Package for the Social Sciences, version 15 (SPSS Inc, Chicago, Ill), was used to calculate descriptive statistics. Mean values, standard deviations, minimum values, maximum values, and 95% confidence intervals (95% CIs) were calculated. One-way analysis of variance (ANOVA) followed by a post-hoc Scheffé test was applied to find differences in shear bond strength between treatments. Differences indicated by P value <5% were interpreted as significant. Additionally, failure types were classified, and the relative frequencies of failure mode in each tested group were computed, together with the corresponding 95% CI.[23]

RESULTS

A box-plot description of bracket shear bond strength (Figure 1) for the differently prepared enamel surfaces is given in Table 2. In Table 3, mean values and confidence intervals are depicted.

Bracket bond strength was significantly reduced on demineralization as compared with sound enamel ($P <$ .001), when the enamel did not receive further pretreatment. Fluoride pretreatment of demineralized enamel with Elmex Gelee or Clinpro did not increase bond strength as compared with nonpretreated demineralized samples (Elmex, $P = .903$; Clinpro, $P = .085$). Thereby, the two fluoride regimens did not statistically significantly differ from each other ($P = .445$). However, application of Icon after demineralization led to significantly better adhesion of the brackets as compared with both fluoridation methods ($P < .001$). Nevertheless, pretreatment of the demineralized enamel surface with Icon resulted in significantly lower

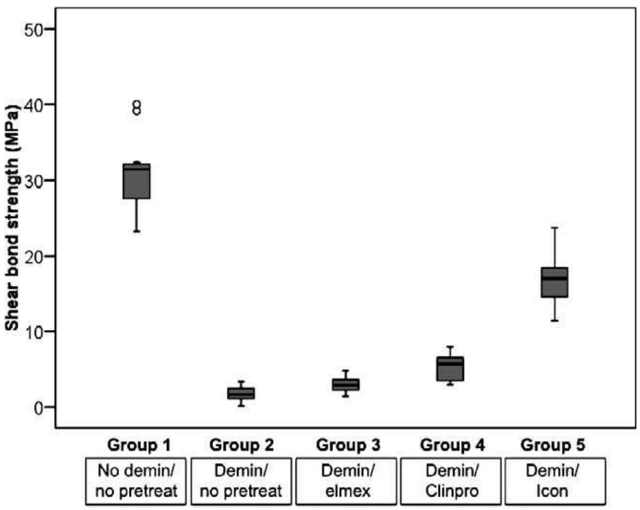

Figure 1. Box-plot description of shear bond strength in the experimental groups.

bond strength of the brackets compared with application of the brackets on sound, nonpretreated enamel ($P < .001$).

Table 4 presents observed frequencies among different types of failures observed after debonding. All fractures in groups 3 and 5 were of the adhesive type. In groups 1, 2, and 4, each 10 adhesive failures and 2 cohesive in enamel were recorded. Neither cohesive failures in cement/resin nor mixed failures were observed.

DISCUSSION

In the present investigation, the shear bond strength of brackets was tested on demineralized bovine enamel. Demineralization of the enamel was conducted using the protocol described by Buskes et al.[21] This protocol has proved to produce initial enamel lesions with very similar characteristics, as are found in natural initial lesions, although artificial lesions so created are less deep. An advantage of using artificial lesions is that these lesions are better standardized than natural lesions in human teeth. This is especially true when bovine teeth are used, and high homogeneity is seen among different teeth. Previous studies have shown that shear bond strength measurements of different substrates revealed equal or, depending on the adhesives tested, only slightly different values between bovine and human enamel.[24–26] Bovine teeth are also widely used in adhesion, as well as in resin infiltration tests.[27,28]

Artificial enamel lesions were created following studies investigating resin infiltration in vitro and exhibiting the typical histologic structure of enamel caries (intact surface layer, lesion body, demineralization front).[10,27,29–31]

According to these considerations, bovine enamel with artificial carious lesions was taken in the present study as a substitute for testing bracket bond strength to demineralized human enamel. After pretreatment with the infiltrating resin or fluorides, respectively, application of the brackets was done after a time lapse of 56 hours, during which time the samples were stored in artificial saliva. The saliva substitute used has proved able to induce remineralization of initial carious lesions.[32]

The time lapse between pretreatment and bracket application was chosen to allow remineralization supported by fluoride application, and to simulate clinical conditions, in which rebonding of a bracket might not be feasible immediately in the same session when the patient appeared with this event. One might also argue that adhesive fixation of brackets to freshly fluoridated enamel might be impaired as the result of hampered efficacy of phosphoric acid etching. However, some studies have shown controversial results regarding the negative effects of topical application of fluoride on bracket bond strength.[33–36]

Thus, it was decided to postpone bracket application to fluoridated enamel to allow some dissolution of the applied fluoride layer. For standardization of the protocol, bracket application to the samples pretreated with the infiltrating resin was carried out until after 56 hours storage in artificial saliva. Mandibular incisor brackets were used because of their flat bases, thus ensuring optimal adaptation to the tooth surface. It should be noted that when brackets are more curved, mismatch between the curvature of the bracket base and the tooth surface will be possible. This mismatch will affect the stress distribution between the adhesive cement and the tooth surface. Thus, in the present study, the film thickness was standardized by placing the brackets under a load of 500 g.

The present study showed that shear bond strength was reduced when brackets were applied to demineralized enamel. No significant improvement in shear bond strength was recorded when the demineralized

Table 2. Box-Plot Description of Shear Bond Strength in Experimental Groups

Group 1 (control; no demineralization/no pretreatment)	Without demineralization, without enamel pretreatment
Group 2 (demineralization/no pretreatment)	With demineralization, without enamel pretreatment
Group 3 (demineralization/Elmex gelee)	With demineralization and pretreatment with fluoride/Elmex gelee
Group 4 (demineralization/Clinpro varnish)	With demineralization and pretreatment with fluoride/Clinpro varnish
Group 5 (demineralization/Icon resin)	With demineralization and pretreatment with infiltrating Icon resin

Table 3. Mean (Standard Deviation), Minimum, Maximum, and 95% Confidence Interval (95% CI) of Shear Bond Strength, MPa

Group	Mean*	Minimum	Maximum	95% CI
1. Control	30.8 (5.1)[c]	23.2	40.0	(27.5, 34.0)
2. No pretreatment	1.8 (1.0)[a]	0.2	3.4	(1.1, 2.5)
3. Elmex	3.0 (1.0)[a]	1.4	4.8	(2.3, 3.6)
4. Clinpro	5.3 (1.7)[a]	2.9	8.0	(4.1, 6.3)
5. Icon	17.0 (3.4)[b]	11.4	23.8	(14.8, 19.2)

* Different letters—a, b, and c—represent a significant difference in post-hoc test.

Table 4. Relative Frequencies (%) of Different Failure Modes With 95% Confidence Interval in Parentheses

Group	Adhesive (95% CI)	Cohesive in Cement/Resin	Cohesive in Enamel (95% CI)	Mixed
1. Control	83 (52, 98)	–	17 (2, 49)	–
2. No pretreatment	83 (52, 98)	–	17 (2, 49)	–
3. Elmex	100 (73, 100)	–	–	–
4. Clinpro	83 (52, 98)	–	17 (2, 49)	–
5. Icon	100 (73, 100)	–	–	–

enamel was fluoridated before bracket placement. An explanation for this result might be found in a recent study,[37] in which decreased penetration of bonding agents into demineralized enamel was noted, when fluoride agents had been applied before bonding. This disruption of the bonding agent could be a cause of the reduction in shear bond strength.

Demineralized samples were treated with the caries infiltrating system or with fluorides before conventional adhesive was used before bracketing, in keeping with manufacturers' recommendations. Thus, etching in the infiltrating resin group was performed with 15% HCl. Previous studies by Paris et al.[38,39] demonstrated that the surface layer of caries lesions can be eroded almost completely by 15% hydrochloric acid compared with 35% phosphoric acid, allowing for better resin penetration. Therefore, not only the resin itself but also the type of etching might influence shear bond strength. However, it has to be considered that natural enamel lesions might be deeper than the artificial lesions created in the present study (~100 micron), which might lead to incomplete penetration and reduced bond strength under clinical conditions.

In the present study, an attempt was made to evaluate failure types occurring on the bracket surface. After debonding, when bracket bases were evaluated, in all groups the composite layer on the bracket was found to be predominantly intact, indicating that the adhesive cement was well attached to the bracket. This fact shows that the problem zone of strength was inside the demineralized enamel. To estimate the results of the present study, it should be noted that an outstanding significantly higher penetration of resins into caries-like lesions can be expected when the infiltrating resin is used as compared with conventional adhesive systems.[7] Thus, it can be assumed that with infiltrating resin, pore volumes had been occluded so that adhesion of the bracket could be performed properly.

CONCLUSIONS

- The null hypothesis has to be partially rejected, in so far as pretreatment with an infiltrating resin is able to improve the bond strength of brackets to demineralized enamel, but not pretreatment with fluoride regimens.
- Nevertheless, because of limitations in the study design (in vitro study, artificial lesions), these effects should be evaluated in clinical studies as well.

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